Hot Water Extraction of Soil Nutrient Elements

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Introduction

For a long time there has been a great need for using water in routine soil extraction methods. Water has been tried in the determination of available nutrient content of soils since the middle of the last century, but the analytical measurement of nutrients was difficult because of water's low solvent power. In the past decades several methods were introduced to increase the solvent power of water or to increase the speed of the process. At the beginning of this century, KÖNIG (1906) autoclaved soil samples in water vapour at 400-500 kPa pressure. This method was good, especially for potassium. Later, hot water extraction was used in the determination of boron, when the soil-water suspension was boiled for a few minutes (BERGER & TRUOG, 1944). AMER (1955) used anion exchanger resin in addition to water to reach a total desorption of phosphorus. PAAUW (1969) increased the efficiency of extraction by increasing the soil:water ratio to 1:50 in the case of phosphorus. NÉMETH (1976) developed the electro ultrafiltration (EUF) method and instrument suitable for serial analyses. The EUF method is a water extraction method directed by electric field and accelerated by vacuum. The extraction is undertaken at 20 °C and 80 °C. KÖRSCHENS et al. (1984) used hot water for the measurement of available C and N content of soils, in a Soxhlet extraction process technically similar to measurement of lipids. SUNTHEIM & MATZEL (1985) applied a continuous water extraction method (KWE) of simpler technical means during which water is filtered with constant speed through a soil-sand mixture, and the amount of phosphorus is determined from the extract. The EUF method is also suitable for determining the kinetics of release of nutrients into solution (FÜLEKY, 1987).

The aim of our work was to introduce the principle of the „espresso coffee maker" when developing a soil extraction method rapid and suitable for routine analyses, i. e. to use high temperature and pressure to increase the speed and efficiency of the extraction.

Materials and Methods

36 characteristic Hungarian soil types were used in the laboratory work. Their principle physical and chemical characteristics covered a wide range (Table 1).

Table 1

Some chemical and physical characteristics of the soils used

Characteristic	X_{min}	X_{max}	$X_{average}$
$pH_{(KCl)}$	3.8	7.8	5.8
$CaCO_3$, %	0.0	27.4	2.5
Humus, %	0.2	7.4	2.6
Sand, %	1.6	94.7	27.2
Clay, %	2.5	53.3	23.4
Smectite + Vermiculite, %	0.0	64.1	20.9

To compare the results 0.01 *M* $CaCl_2$ soluble NO_3-N and NH_4-N (HOUBA et al, 1987), AL (ammonium lactate soluble) -P, -K, -Ca, -Mg, -Na content (EGNER et al, 1960). EDTA soluble Fe, Mn, Zn, Cu content, using 25 g soil and 50 cm^3 0.1 *M* KCl and 0.05 *M* EDTA extractant with 2 hours extraction time, EUF-K (FÜLEKY, 1987), water-soluble P (PAAUW, 1969) and Olsen-P (WATANABE & OLSEN, 1965) were determined. To compare the results with plant uptake of nutrients a pot experiment was carried out with the 36 soils and rye-grass as test plant. In the pot experiment the amounts of P and K taken up in 6 subsequent cuts of the shoots were measured. Another pot experiment was set up using sunflower on 16 soils chosen from the 36 soils. N, P, K, Ca, Mg, Fe, Mn, Zn, Cu content in above-ground parts of 4-week-old plants were determined.

Hot water percolation (HWP) was carried out in an „espresso coffee maker"-like instrument. The changeable sample holder contained 40 g soil, a mixture of 30 g soil/10 g sand. Water preheated to 102-105 °C in the container was pressurized at 120-150 kPa pressure through the sample. Water leaching through the soil was collected in 100 cm^3 aliquots, and the percolation time was measured. In the extract NO_3-N, NH_4-N, P, K, Ca, Mg, Fe, Zn and Cu contents were determined by titration, after steam distillation, spectrophotometry, flame photometry and atomic absorption spectrophotometry, respectively. The extractions and determinations were made in 5 replications.

Results

The time of percolation of 100 cm^3 extract through the soil was 41 min, while an extract of 5x100 cm^3 was obtained during 188 min on the average, respectively. Hence it was necessary to dilute the soil with sand. However,

when using 50% soil – 50% sand mixture the percolation was too fast: 100 cm^3 extract was obtained in 0.8 min, and 500 cm^3 during 3.6 min on the average. To increase the effectivity of the percolation the amount of sand in the prepared sample was reduced to 25%, and whence a suitable percolation rate and more effective extraction resulted. When using the 75% soil:25% sand ratio, the first 100 cm^3 aliquot was obtained in averagely 2.6 min, while the average time for obtaining the 500 cm^3 extract was 11 min. These periods might increase significantly when using soils with high clay (especially smectite) content, because of the significant swelling of the soil. In such soils the ratio of sand in the mixture had to be increased.

Table 2
Variability of the HWP method

Fraction	1 fraction				1-5 fraction			
	X_{min}	X_{max}	X_{aver}	CV%	X_{min}	X_{max}	X_{aver}	CV%
NH_4-N	5.5	38.0	13.7	11.0	8.1	77.1	28.7	12.0
NO_3-N	0.7	70.7	12.9	12.0	2.6	77.3	17.8	11.0
K	13.9	123.8	47.5	4.9	33.1	329.4	107.3	6.0
P	0.2	24.0	4.7	9.5	0.8	94.5	18.3	5.0
Ca	11.9	250.0	97.4	6.1	11.9	413.2	189.0	7.0
Mg	0.1	30.8	14.8	5.0	0.1	60.8	20.7	6.0
Na	2.8	59.7	28.4	3.5	3.1	76.7	36.0	5.0
Fe	0.0	6.1	2.0	24.0	0.0	26.8	4.1	24.0
Cu	0.1	11.5	3.1	13.0	0.3	26.6	6.9	15.0
Mn	0.0	11.4	2.6	20.0	0.0	17.1	4.3	17.0
Zn	0.1	15.0	3.3	15.0	0.1	19.8	4.7	14.0

The investigations were made with soil:sand mixtures of 75%:25% ratio. We have examined the reproducibility of the method. Table 2 shows the lowest and highest values of NH_4-N, NO_3-N, K, P, Ca, Mg, Na, Fe, Mn, Zn, Cu contents measured by the HWP method in 36 soils. The average values and the average % deviation for the individual elements (presented as the average of the CV % values determined for the different soils) are also given in Table 2. It can be seen that the majority of nutrients can be extracted in detectable amounts by this method. The method indicates the differences in the nutrient content of different soils (x_{min}-x_{max}) in a satisfactory manner. Reproducibility of the method is generally <10%.

The element content obtained by this method were compared with the results of conventional methods. Table 3 shows values obtained by conventional methods. The element contents determined by the HWP method and by the conventional methods are generally of the same order of magnitude: the HWP method generally gives higher NH_4-N values than the 0.01 *M* $CaCl_2$ method,

Table 3

The conventional methods used and their linear correlations with the HWP method

	X_{min}	X_{max}	$X_{average}$	$R_{1\ fraction}$	$R_{1\text{-}5\ fraction}$
$CaCl_2$-NH_4-N	3.8	30.0	8.8	0.7907	0.7841
$CaCl_2$-NO_3-N	0.7	89.0	15.0	0.6290	0.7059
AL-P	16.0	349.0	88.6	0.7514	0.7851
OLS-P	4.9	166.0	37.7	0.8636	0.8927
H_2O-P	0.4	24.3	5.1	0.8543	0.8624
AL-K	42.0	780.0	206.6	0.7832	0.8540
EUF-K	62.0	514.0	203.3	0.7963	0.8714
EDTA-Ca	163.1	8776.7	5016.0	0.2610	0.4320
EDTA-Mg	13.1	395.9	195.0	0.5968	0.6122
EDTA-Fe	16.2	452.2	286.3	0.8367	0.8826
EDTA-Mn	37.5	552.4	252.5	0.4374	0.5410
EDTA-Zn	0.7	17.6	3.8	0.5230	0.5969
EDTA-Cu	0.5	29.3	4.8	0.0293	0.0506
AL-Na	1.0	93.0	37.5	0.8658	0.8667

about the same NO_3-N content and H_2O P-content, while AL-K and EUF-K extracts a little more K, and Olsen-P a little more P, than the HWP method.

Considering the importance of using the HWP method in advisory systems, its close correlation with results of conventional methods is even more important. The method shows a very close correlation in the case of NH_4-N, AL-P, Olsen-P, H_2O-P, AL-K, EDTA-Fe and AL-Na, a fairly close correlation in the case of NO_3-N, EDTA-Mg and EDTA-Zn, while only a slight correlation in the case of other elements.

It is very important that these correlations were very close in respect to the most important macroelements. The correlation of the method with plant nutrient uptake was also investigated. There is a very close correlation with the phosphorus uptake of both sunflower and ryegrass plants; there is also a very close correlation with Mn uptake, and close correlation between the NO_3-N and NH_4 content and plant N uptake. In addition there is a close correlation with the K uptake of ryegrass plants, and there are also correlations with the K, Ca uptake and Cu uptake of sunflower. There are no correlations between the Fe and Zn values measured by the HWP method and the Fe and Zn uptake of sunflower plants.

We have examined the possibilities of a mathematical presention of the results obtained by HWP method. For each element there was a close correlation between the amount of percolated water and the extracted amount of elements. This relationship is described in a satisfactory manner in each case by the saturation equation $y = A(1\text{-}e^{-kV})$ where V = the amount of percolated water. The first-order kinetic equation $y = A(1\text{-}e^{-kt})$ is suitable for the formal kinetic de-

scription of the release processes of nutrients during HWP extraction, where t = time. With the help of this latter equation the speed of release into solution can be determined at any time of the percolation. For example, in soil No. 3, after 100 s percolation time the speed of release of NH_4-N into the solution was $216 \cdot 10^{-1}$ mg N kg^{-1} s^{-1}, while in soil No. 23 it was $12 \cdot 10^{-3}$ mg N kg^{-1} s^{-1}.

In accordance with the last equations there is a close and mathematical relationship between the elemental content released into the first 100 cm^3 extract during the percolation process and the elemental content of the later fractions. Results shown in Table 3 also indicate that for advisory purposes it is quite adequate to collect the first 100 cm^3 extract and to determine its elemental composition.

Conclusions

The major advantages of the method are:

– the extraction can be carried out rapidly and the quantity of extracted elements can be measured continuously;

– the simple, cheap apparatus uses a cheap extracting agent, distilled water, which also justifies the theory;

– a number of elements can be determined from a single extract;

– the method can be automated and is suitable for serial analyses, but the extraction can also be carried out on farms.

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